

# ACTIVITY REPORT

December 2002



**Natural  
Gas &  
Oil  
Technology  
Partnership**

bringing department of energy national laboratories capabilities to the petroleum industry

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Note: Natural Gas and Oil Technology Partnership projects are reported according to the following schedule:

**January, March, May, July, September, November**  
Drilling, Completion, and Stimulation Technology  
Oil and Gas Recovery Technology  
Diagnostic and Imaging Technology

**February, April, June, August, October, December**  
Upstream Environmental Technology  
Downstream Environmental Technology  
Natural Gas Technology

**Natural Gas and Oil Technology Partnership on the World Wide Web: <http://www.sandia.gov/ngotp/>**

## Upstream Environmental Technology

### Ecological Framework to Evaluate the Effect of Size and Distribution of Releases at Upstream Petroleum Sites

(American Petroleum Institute, BP Amoco, ChevronTexaco, ExxonMobil, Gas Technology Institute, Unocal, LLNL, and ORNL)

#### Highlights:

- Modeling of American badger and prairie vole at Tallgrass Prairie Preserve continues.
- Four manuscripts in progress.

ORNL made a number of improvements to the landscape model for the American badger. Researchers now permit less movement during specified months (typical of hibernation). In addition, researchers calculated the proportion of females that find mates to help determine the role of Allee effects caused by habitat fragmentation, generalized the calculation of habitat-related survival, and added age-specific mortality rates. Simulations of landscapes with different amounts of area covered by brine were repeated and show a pattern of a reduced final population size and likelihood of persistence as the spill area increases. However, the actual area impacted in the Tallgrass Prairie Preserve (TPP) is much smaller than those causing a reduced population size in the simulations. A manuscript on the badger model is being prepared, as well as a manuscript describing two statistical models for generating spills. Project researchers are currently fitting a gamma model to the actual spills in the TPP.

LLNL made several improvements to the vole model. The bioenergetic module was reparameterized with more realistic values from the literature. The rule governing movement of voles between cells was revised so that wandering animals would choose a cell with the highest amount of biomass from among adjacent cells. In addition, if two wandering animals reached an empty cell at the same time, the largest animal would become resident. The effects of predation and dispersal were also included in the model. Predation was included through the removal of animals from the population at a set rate (which can be varied), with wandering animals experiencing a higher level of predation than resident animals. Dispersal through the site boundary was included, which also results in the loss of animals from the population.

Simulations are being conducted to further evaluate the time to extinction based on area size and level of predation. The fragmentation simulations are being designed and will be conducted to correspond with simulation work being done by ORNL using the badger model. A manuscript is being prepared on the vole model to be submitted to an appropriate ecological modeling journal.

ORNL and LLNL are preparing a manuscript summarizing the ecological framework developed in this project. This manuscript will be published as a chapter in an American Society of Testing and Materials (ASTM) Special Technical Publication resulting from a symposium on Landscape Ecology and Wildlife Habitat Evaluation: Critical Information for Ecological Risk Assessment, Land-Use Management Activities, and Biodiversity Enhancement Practices to be held in Kansas City, Missouri April 1–9, 2003.

**Estimation and Reduction of Air Quality Modeling Uncertainties** (Envair, EPRI, and LBNL)

Improved emissions tables for the August 1990 ozone episode in the SARMAP domain were obtained from the Marr and Harley. The improved emissions are for motor vehicle (mobile) sources only and are derived with the aid of fuel sales data. These revised emissions will be used instead of CARB emission estimates. An interface program was written that processes these emissions from point, area, biogenic, and mobile sources, and produces CMAQ-ready input emissions files at a grid resolution of 4 km x 4 km.

The draft doctoral dissertation manuscript concerned with the use of models and uncertainty in urban- and regional-scale air quality management based on a case study of central California planning in the 1990s was completed. Chapters about the theoretical responses to the challenges of air quality planning and the limitations of photochemical air quality models (Chapter II), the SARMAP modeling application used in the case (Chapter VIII), a summary of conclusions (Chapter XII), and recommendations (Chapter XIII) were drafted.

Researchers completed a draft of a critical review paper about the evaluation of uncertainties in air quality simulation models used for regulatory compliance that will be submitted to the Annual Review of Energy and Resources.

**Remote Sensing for Environmental Baseline and Monitoring**(ChevronTexaco,  
UC-Davis, and ORNL)**Highlights:**

- Report on analyzed field data from the Jornada Experimental Range completed.
- Remote sensing proposed in Osage county, OK.

In April 2002, ORNL scientists received data for 665 measurements that were made earlier at the Jornada Experimental Range (JER) by Agricultural Research Service (ARS) investigators. In September, ORNL completed a report on the analysis of field data from the JER. The goal of the analysis of the field data was to explore the resolution limits for pixel unmixing and plant species identification. The approach was to partition hyperspectral measurements into distinct groups that correspond to different stress states or species and use the distinct groups (or endmembers) as a basis for the measurements.

As the research progressed, ORNL scientists created a linearly independent basis for a set of measurements and realized that there can be many more endmembers than basis vectors (for the ARS data set, there are four basis vectors and 20 endmembers). Endmembers are neighborhoods in an N dimensional space. A corollary is that it is not possible to do pixel unmixing (one measurement in a 4D space cannot determine the fractions of each of 20 endmembers that are present in the measurement).

In FY 2003, ORNL and LLNL scientists propose to collect hyperspectral remote data in selected regions in Osage county, OK, and analyze the hyperspectral imagery to detect brine scars, oil pits, and plant stress associated with brine and oil. Osage county is on the Osage Indian Reservation and has been a major oil producing area (38,500 oil wells), since 1896. The county is large (2,260 square miles) and 1,480 square miles are within a quarter mile of an oil well. The mineral rights are owned by the Osage Nation, and there are 500 independent operators in the county. Many areas in the county have brine scars or weathered oil pits. Collaborators on the project will include the US Geological Survey (USGS) and the ARS.

**Modeling of Water-Soluble Organic Content of Produced Water**(ChevronTexaco,  
ConocoPhillips, Shell, Statoil, and ORNL)**Highlight:**

- Project researchers presented at the Produced Water Conference.

The Groundwater Protection Council (GWPC) sponsored a conference on produced water in Colorado Springs, October 16–17. Beneficial use of produced water is of interest to many Western states as it would simultaneously deal with the problems of water shortage caused by drought conditions and the large volume of waste water generated by coal bed methane and petroleum production. Although the final disposal of produced water on-shore is different from wells in the Gulf of Mexico, many of the issues of contamination and remediation are similar. Hence, ORNL attended the conference to present the effort on produced water modeling and characterization and to learn about research and approaches developed elsewhere in the industry and government. The conference attracted attendees from many of the Petroleum Environmental Research Forum (PERF) partnership industries, the Department of Energy, state representatives, national labs, and universities.

Many topics were discussed at the conference: regulations, pilot projects, technologies for remediation, and technical approaches. The primary disposal methods used to handle water produced on-shore are by re-injection and evaporation. Discharge into waterways can only be done after the water is cleaned to certain environmental standards set by the Environmental Protection Agency, the state, or customers for the water. Removal of inorganic compounds (e.g., salt) is usually done using reverse osmosis (RO), an expensive technique that is effective but very sensitive to organic fouling. Hence, pre-treatment to remove oil and grease is an important consideration for RO. This removal of organics is where the methodology currently being pursued at ORNL may contribute to the remediation of on-shore as well as offshore produced water.

Project researchers presented the collaborative Petroleum Environmental Research Forum (PERF) project on characterization and modeling of produced water at the conference.

**Science-Based Methods to Assess Risks Attributable to Petroleum Residues Transferred from Soil to Vegetation**(ChevronTexaco,  
Petroleum Environmental Research Forum,  
UC-Berkeley, UC-Davis, and LBNL)**Highlights:**

- Poster of year-one progress presented at proposal review meeting in Houston, TX.
- Chamber experiments designed to optimize exposure conditions begun.

Researchers from LBNL prepared and presented a poster describing progress during the first year of the project at the Upstream Environmental Program proposal review meeting in Houston, TX. Researchers continue to work on a manuscript describing the literature review and development of the revised plant uptake model for petroleum hydrocarbons.

Experiments are under way using wheat and agricultural soils in the growth chambers to test various combinations of soil, planting and growing conditions for the plant uptake exposure studies. Preliminary results indicate that a mixture of two parts autoclaved agricultural soil to one part sand provides a well-drained weed-free mixture for growing the wheat in pots. An air monitoring system was constructed and is being tested by researchers at University of California-Davis for use in the chamber to characterize gas and particle phase concentrations during the exposure events.

## Interactive Information System on Drilling Waste Management Practices (ChevronTexaco, Marathon, ANL)

### Highlights:

- Developed prototypes of the homepage format and two modules.
- Collected extensive information on state regulatory practices.

There are numerous practices employed by the oil and gas industry for managing drilling wastes. These practices are governed by geographic, climatic, and environmental restrictions. Within the United States, there is no single set of national standards or guidelines for drilling waste management, rather each state has developed its own set of requirements to protect human health and the environment. Internationally, the picture becomes even more complicated, as various countries with established and developing oil and gas industries have different drilling waste management requirements and, in some cases, no specific requirements. ANL, Marathon, and ChevronTexaco are working to develop an interactive web-based information system that provides information on the technical and economic aspects of drilling waste practices and a review of up-to-date state and federal regulations on waste management.

The website will include components such as a technology description module to familiarize readers with available options, a regulatory module to summarize existing state and federal regulations governing drilling waste, a case study module to exhibit successful implementations of various technologies, and an interactive technology identification module to help users identify waste management options applicable in their region. This information system will provide operators with easy access to regulations, waste management options, technology, and cost data, thus allowing them to choose the option with the most environmental and economic benefits. Such a website could benefit both small companies in the United States and larger companies that operate internationally.

The team collected information relating to drilling waste management options and regulatory requirements. In the fall of 2002, ANL developed a framework for the web-based information system along with prototypes of the technology description and regulatory modules.

### Publications and Presentations

Veil, J.A., "Drilling Waste Management: Past, Present, and Future," *SPE* 77388, SPE Annual Technical Conference and Exhibition, San Antonio, TX, September 29-October 2, 2002.

Fidler, B., "Interactive Website for Drilling Waste Management," 9th International Petroleum Environmental Conference, Albuquerque, NM, October 22-25, 2002

Puder, M.G., B. Bryson, and J.A. Veil, draft "Compendium of Regulatory Requirements Governing Underground Injection of Drilling Wastes," prepared by Argonne National Laboratory for the U.S. Department of Energy, Office of Fossil Energy, National Petroleum Technology Office, November 2002.

## Downstream Environmental Technology

### Kinetics of Biochemical Upgrading of Petroleum (Biocat, ChevronTexaco, Shell, and BNL)

No report received.

## **A Predictive Model of Indoor Concentrations of Outdoor PM<sub>2.5</sub> in Homes**

(Aerosol Dynamics,  
Western States Petroleum Association, and LBNL)

A paper entitled “The Use of Time- and Chemically-Resolved Particulate Data to Characterize the Infiltration of Outdoor PM-2.5 into a Residence in the San Joaquin Valley” was submitted to *Environmental Science and Technology* in December, 2002.

Progress was made on development and testing of the transient model of indoor aerosol concentrations of outdoor origin. The transient model was used in a regression analysis to determine best-fit penetration and deposition rates for nitrate, sulfate, and total carbon constituents of indoor aerosols. The deviations of the model from the measured indoor aerosol concentrations are examined for statistically significant correlations existing between the deviations and building operating conditions, site meteorology, and concentrations of gas phase ammonia and nitric acid. A report summarizing these findings is being prepared.

Progress was made on the development of the model to describe the transformation of ammonium nitrate aerosol inside the house. The model combined the infiltration rate of the particulate, the evaporation rate of the volatile aerosol and the loss rate of the gaseous products due to sorption to the walls. The work will result in a transient model for nitrate aerosol vaporization that will be compared with measured concentrations of ammonium nitrate aerosol, and gaseous nitric acid and ammonia. It will be incorporated as a submodel into the overall transient infiltration model.

A draft manuscript describing the characterization of the behavior of carbonaceous aerosols (of outdoor origin) in residences is being prepared. It contains a comparison of the results obtained by several different instruments that characterize either organic carbon, black carbon, or both. The paper also focuses on the artifacts associated with the individual carbonaceous aerosol measurement methods. Project researchers are using the measurement results associated with outdoor/indoor concentrations of carbonaceous aerosols of outdoor origin to understand infiltration and loss mechanisms in residences.

## **A Predictive Model of Indoor Concentrations of Outdoor Volatile Organic Compounds in Homes**

(American Petroleum Institute,  
Western States Petroleum Association, and LBNL)

Project researchers added a third physical-conceptual model for describing sorption to the GUI-based code, which is designed to find the best-fit adsorption and desorption parameter values for user-input experimental data. This new model includes two sinks - one at the surface and one in the bulk material - with separate transfer rates to and from the deeper sink. This mathematical formulation can be used to represent a variety of physical/chemical processes, for example, diffusion followed by sorption and for a change in the energy state of sorption at the surface, chemical reaction. The model was applied to four experiments in a 50-m<sup>3</sup> chamber under three different levels of realistic furnishings.

Project researchers also initiated modeling work to link experimental results to past work that characterized sorption interactions of volatile organic compounds (VOC) on individual materials. Specifically, researchers ran the full indoor dynamics model with the actual surface to volume ratios of all materials in the chamber and used as input the adsorption and desorption rates obtained by Won et al. for individual VOC-material combinations. The model was then run with emissions and ventilation patterns that matched experimental conditions. The scaled-up model predicted concentrations of toluene and ethylbenzene will be compared to experimentally measured concentration profiles of the same compounds.

Researchers completed two more experiments to characterize adsorption, desorption, and equilibrium partitioning rates in a room-sized chamber constructed of common residential materials. These new experiments compliment earlier experiments conducted while the chamber was filled with common indoor furnishings such as upholstered chairs, cotton draperies, and wood/veneer furniture. In the first new experiment, all furnishings were removed, leaving the carpet/padding system and wallboard as the only materials in the room. In the second new experiment, the carpet was also removed, leaving only wallboard. Both these and the earlier two experiments were analyzed using the code described above to obtain sorption and equilibrium partitioning rates for the various conditions.

### **Developing Enzyme and Biomimetic Catalysts for Upgrading Heavy Crudes via Biological Hydrogenation and Hydrodesulfurization** (ChevronTexaco, ANL, and ORNL)

#### **Highlight:**

- Conducted preliminary characterization (molecular weight and pI) of nickel-iron active center peptide complex derived from *D. gigas* hydrogenase.

This project investigates the potential of enzymatic and biomimetic catalysts for hydrogenation of oil compounds with the goal of upgrading crudes via sulfur removal and potentially molecular weight reduction.

Development of the nickel-iron (Ni-Fe) active center peptide complex is under way. Further purification of the complex was conducted via chromatographic and membrane separation techniques. This also enabled preliminary characterization of the molecular weight and isoelectric point of the complex. Size exclusion chromatography shows that the size of the complex is about 1300 Da and ion exchange chromatography suggests that the pI of the complex is approximately 7.0. Further experiments are needed to confirm the pI value. These results match the predictions based on the estimated structure obtained from protein digestion.

The hydrogen uptake activity of the complex was used to follow the development of the purification protocol. The complex has significantly higher activity at 60°C than 30°C and therefore has allowed us to work with very small quantities of the complex. It was found that the native enzyme is also thermostable for several days at 60°C (as measured by hydrogen uptake activity). Further experiments are being conducted with the native enzyme and the complex simultaneously.

The hydrogen evolution activity of the crude preparation was also assessed at various temperatures. Preliminary results indicate that the complex is stable and active for several days at temperatures up to 60°C. The activity of the complex was also compared with the native enzyme, which also had activity up to 60°C. The comparison between the two catalysts showed that the Ni-Fe active center complex was at least as thermostable as the native enzyme as measured by hydrogen evolution activity. Specific activity of the complex is not yet known but experiments to purify it to homogeneity are under way.

### **Characterization and Reaction Behavior of Sterically-Hindered Sulfur Compounds in Heavy Crudes with Nano-Sized Molybdenum Disulfide** (ChevronTexaco, BNL, and ANL)

#### **Highlight:**

- Project researchers continue to synthesize and characterize nanomaterials.

Project researchers continue to synthesize and characterize nanomaterials. Another sample was prepared by sonolysis of commercial micron-sized molybdenum disulfide (MoS<sub>2</sub>) obtained from Aldrich. The commercial micron-sized MoS<sub>2</sub> serves as a baseline. Four samples now form the project inventory of potential hydrodesulfurization (HDS) catalysts: the sonicated commercial micron-sized MoS<sub>2</sub> and three previously synthesized nano samples, unsupported MoS<sub>2</sub>, unsupported Co/MoS<sub>2</sub>, and MoS<sub>2</sub> supported on gamma-Al<sub>2</sub>O<sub>3</sub>. A detailed characterization of nanosized materials presents a challenge because of their size.

The x-ray diffraction (XRD) studies are now ongoing at the National Synchrotron Light Source (NSLS) at Beam Line X-7B. The analysis shows that the XRD spectra of nanomaterials are different from their micron-sized counterparts. Complementary data on morphology and particle size of the samples are being collected on a JEOL 2000FX, 200KV transmission electron microscope (TEM), with a 0.16 nm resolution. The XRD data are being compared to the Temperature Programmed Reduction (TPR) and the Temperature Programmed Oxidation (TPO) data at ANL. Together, these data will help researchers understand the structural features of the synthesized nanomaterials.

## Development of a Solid Catalyst Alkylation Process Using Supercritical Fluid Regeneration

(Marathon-Ashland and INEEL)

### Highlight:

- INEEL nominated the supercritical fluid catalyst regeneration technology for an R&D 100 award.

Earlier experimental efforts explored the effect of olefin weight hourly space velocity (OWHSV) on catalyst activity maintenance. OWHSVs between 0.20 and 0.30 hr<sup>-1</sup> at an isoparaffin to olefin ratio of 20:1 were explored to determine optimal conditions for efficient experimentation. At an OWHSV of 0.26 hr<sup>-1</sup>, catalyst activity was maintained above 90% of the initial value for approximately 40 hours, or six reaction/regeneration cycles. This OWHSV will allow approximately six experiments to be conducted per month and will be used in the subsequent optimization experiments. Current experimental efforts are exploring regeneration time requirements.

Dan Ginosar provided a presentation on solid acid alkylation catalyst regeneration chemistry at the AIChE annual meeting in Indianapolis, November 7, 2002.

Experimental efforts are exploring regeneration time requirements at reaction conditions of 0.26 hr<sup>-1</sup> (OWHSV) and an isoparaffin to olefin ratio of 20:1. Work in November demonstrated that the required regeneration time may be shorter than previously thought. Efforts are exploring regeneration times between 5 minutes and 2 hours.

## Biocatalytic Alkane Transformation for Viscosity Reduction

(ChevronTexaco and LBNL)

A menu of biocatalytic agents capable of transforming alkanes to alcohols and acids was developed in a previous NGOTP project (Biological Upgrading of Heavy Oils). In this project, several of the most promising biocatalysts were selected from the menu for further characterization and development. All of the biocatalysts selected for this project harbor enzymes that fall in the well-characterized AlkB family of proteins, but the biocatalysts exhibit a diverse range of activities. The objective of this project is to determine if genetic or physiological properties govern the target specificity of the biocatalyst. Experiments are being conducted to measure the transformation kinetics of alkanes in whole cells and cell extracts to differentiate genetic from physiologic controls in target specificity. The genetic code of the alkane transforming enzymes will be determined and the relationship between the gene code and the enzyme specificity will be investigated.

Biocatalyst IP-9 was positively identified as *Rhodobacter ruber* by 16s RNA sequencing. This biocatalyst was tested for the presence of multiple enzymes using a catabolic inhibition assay. Results to date indicate that the organism harbors one enzyme system that catalyzes all alkanes, rather than multiple enzymes with narrower specific ranges. The enzymes harbored by this biocatalyst are capable of transforming a broad variety of other compounds, including nitriles and chlorinated solvents. The application of this biocatalyst to the oxidation of high molecular weight alkanes is under investigation in cooperation with industrial participants. Patenting and licensing options are under discussion.



**Secondary Organic Aerosol Research**

(Aerosol Dynamics, Western States Petroleum Association, and LBNL)

The aerosol measurement campaign for 2002 was concluded in mid-November, and the instruments were removed from the fields. During the last week of October, portions of the forest near the research site were subjected to controlled burning experiments performed by the Forestry Department of the University of California-Berkeley. This provided a unique opportunity to obtain near source aerosol emissions data for forest fires, which is of growing importance nationally due to new visibility legislation. During the active burning period, researchers were able to obtain filter samples of the emissions for more detailed chemical analysis. Similar filter measurements will be performed during the 2003 sampling season to assist in characterization of the aerosol under normal atmospheric conditions.

Researchers processed the data from the 2002 sampling season, focusing on detailed data quality control and quality analysis. A concerted effort at data analysis, focusing on statistical and time series analysis will begin in the spring of 2003.

A poster describing the observations concerning the nuclei mode formation events was presented at the Berkeley Atmospheric Science Center Conference in December.

**Proton Exchange Reactive Membranes for Conversion of Light Alkanes to Clean Liquid Fuel**

(British Petroleum and INEEL)

Work in November and December focused on experimental system testing and membrane production. Minor modifications to the test system were made to accommodate membrane requirements. A small-scale palladium membrane was built and used in initial system testing. Initial experiments found an internal membrane leak at elevated temperatures. A second membrane is being produced. The palladium membrane will serve as a surrogate for the proton conducting ceramic membrane until the ceramic membrane is available.

## Natural Gas Technology

**Molecular Engineering: Next Generation of Gas Purification Technology** (ChevronTexaco, Virginia Commonwealth U, and BNL)

New FY02 project: Reporting will start three months after the DOE FY02 funding arrives at the laboratories.

**Coil-Tubing-Deployed Hard Rock Thermal Spallation Drill and Cavity Maker** (Nextant, NM Tech, and LANL)

New FY02 project: Reporting will start three months after the DOE FY02 funding arrives at the laboratories.

**Scintillating Fiber Neutron Detectors for Well Logging** (CompuLog, Precision Drilling, Technology Services Group, and PNNL)

A non-disclosure agreement was placed with 391545 Alberta, Ltd. so that PNNL may obtain design details of the M.A.N. well logging tool. This information will be used in MCNP calculations to determine the efficiency of various fiber replacements for the  $^3\text{He}$  tubes. The gamma ray background must be determined. The PNNL neutron-sensitive scintillating fibers can handle a count rate of one million counts per second of combined neutrons and gamma rays.

**225° C MWD Using Silicon-On-Insulator (SOI) Electronics** (Baker Oil Tools, Eagle-Picher, Honeywell SSEC, General Atomics, Noble Engineering, Quartzdyne, and SNL)

New FY02 project: Reporting will start three months after the DOE FY02 funding arrives at the laboratories.